

Supporting Information for "The Impact Of Flocculation on In Situ and Ex Situ Particle Size Measurements by Laser Diffraction"

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Introduction

Text S1 details about manova (multilinear anova) analysis used to determine the relative importance of PSD characteristics on required measurement time. Text S2 details about the measurement principle and procedure of Mastersizer-3000 used to measure PSD.

Text S1: Determining the relative importance of PSD characteristics on required measurement time

To determine the relative importance of various characteristics of the PSD on the required measurement time, a manova analysis (multilinear anova) is performed. Figure S12 and S13 indicate the linear dependencies of modality (uni or bi/multimodal), span value (distribution width), particle size (D_{50}) and location (in or ex situ) with the required sampling time (using the three different thresholds) and with each other.

Next, regression models are used to determine which parameters are the most important for determining required measurement time. We assumed that a parameter is important if it is selected as significant in a regression based prediction model. Next, we deselect unimportant parameters by comparing different regression models with anova. We included interaction between the independent variables (SV, D_{50} , modality and in/ex situ).

The required measurement time depends mostly on: location (in/ex situ) for threshold 1 and 3 and the interaction between D_{50} and location for threshold 2. Other important variables were the interaction between D_{50} and bimodality (threshold 1), the interaction between SV and D_{50} (threshold 2), the interaction between SV and field (threshold 2 and 3), and SV (threshold 3).

Text S2: PSD measurements with Mastersizer-3000

Mastersizer-3000 (Malvern Panalytical Ltd., Malvern, United Kingdom), hereafter referred to as Mastersizer (MS), is a laboratory based instrument which uses laser diffraction to measure particle size distribution. It measures the intensity of scattered light as a laser beam travels through a dispersed particle sample. The Mastersizer measures PSD from 0.01 to 3500 μm utilizing a single optical measurement channel and can therefore detect particles that the LISST accumulates in its last size bins. The dispersed sample moves through the optical bench's measurement region, where the particles are illuminated by

a laser beam. The sample dispersion unit guarantees that particles are fed to the optical bench measurement area at the appropriate concentration and in a stable state of dispersion, to enable accurate, reproducible results. A series of 101 detectors then measure the strength of light scattered by the particles within the sample for both red and blue light wavelengths. Mastersizer was used with a large volume (600 ml) wet dispersion chamber, which has an in-built stirrer and sonication probe that enables further dispersion of particle aggregates (flocs). The stirrer speed can be fixed between 1000 rpm and 3000 rpm and the intensity and duration of ultrasounds can be adjusted by the user.

Ex situ particle size measurements using Mastersizer were performed with three stirrer settings (1000 rpm, 2500 rpm and 2500 rpm with ultrasound). Samples were introduced into the LV dispersion unit until an obscuration range of 3-5% was detected to ensure representative measurement. For sample with low SSC, this obscuration range was not achieved. Similar to the order of settings during ex situ particle size measurements with LISST, a sample was first measured with the first setting (1000 rpm) and consequently with the second setting (2500 rpm). During the third setting, along with 2500 rpm, ultrasound was applied for 60 seconds at 100% intensity to achieve maximum dispersion of the particles. For each setting, 5 measurements were recorded and averaged to form one sample. The raw data from each Mastersizer sample was converted to the corresponding PSD using an in built model based on Mie Theory. The conversion model considered particles as irregular shaped. Similar to LISST, the calculated D50 values were subsequently used to report the effect of storage duration and stirring on the particle size distribution.

Data Set S1.

April 21, 2023, 1:50pm

References

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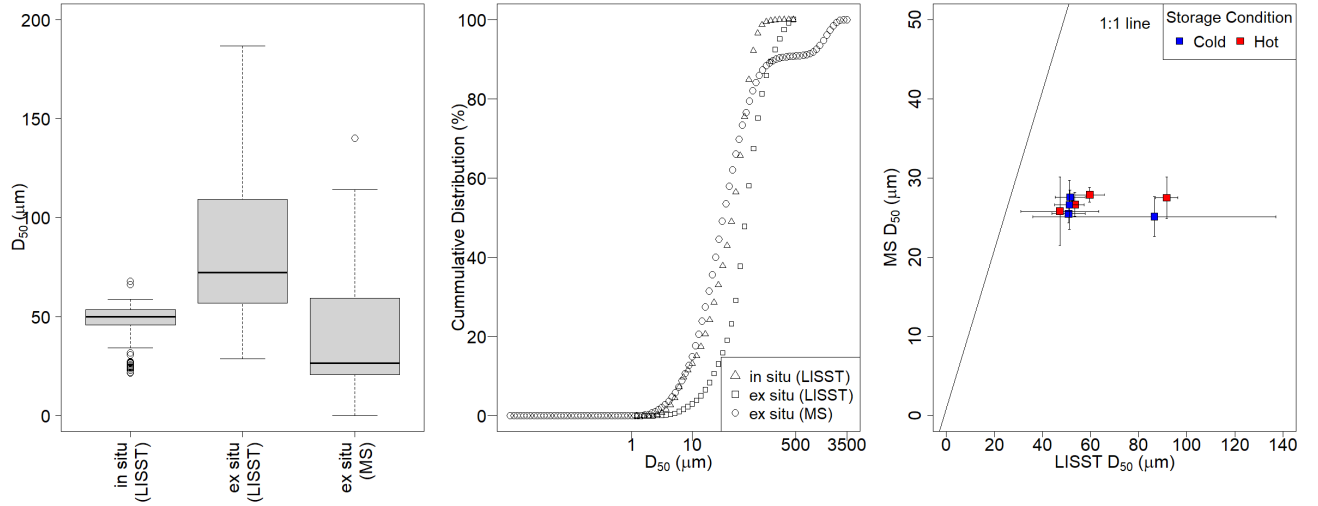


Figure S1. a) Boxplot of median particle size (D_{50}) by sample for all in-situ (LISST) and ex situ (LISST and Mastersizer) measurements collected in Useldange, Luxembourg. The horizontal line in each box represents the median value, and the boxes extend to the interquartile range. Upper and lower whiskers reach the quantiles of 0.975 and 0.025, respectively. The minimum and maximum values are indicated by circles. b) Comparison of in situ and ex situ cumulative distributions by volume for all the samples and recorded measurements. c) Comparison of D_{50} measured ex situ by LISST and Mastersizer.

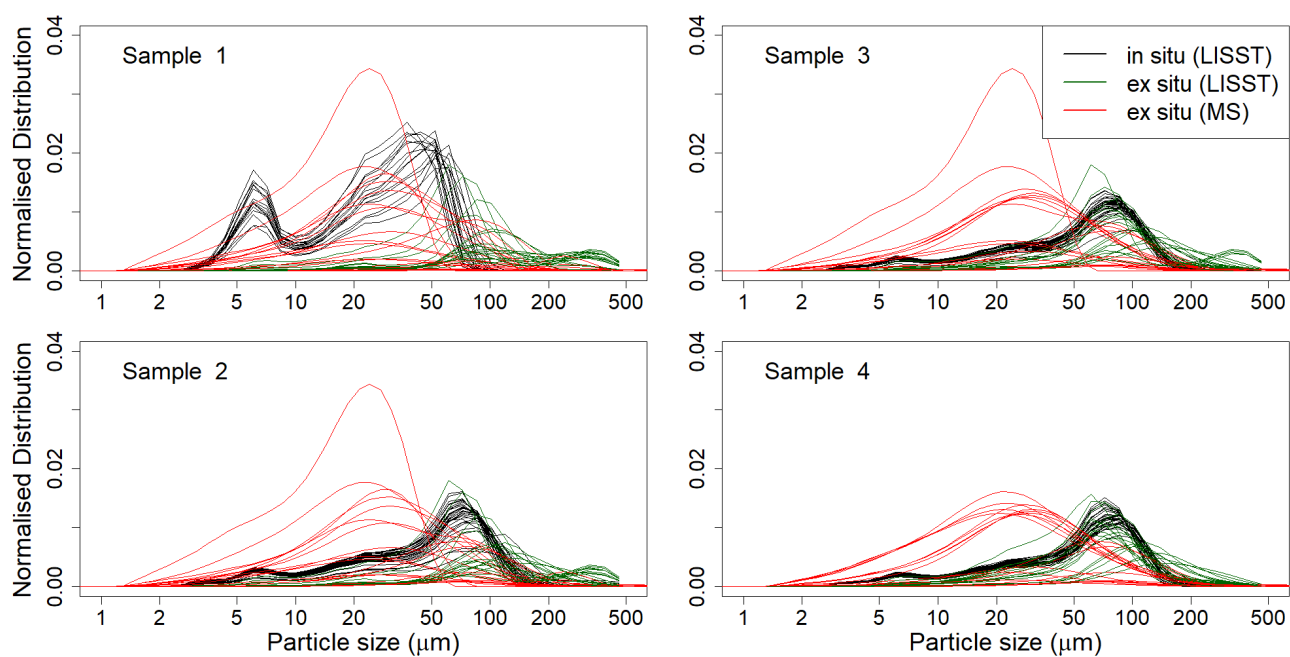


Figure S2. In situ and ex situ particle size distributions of 4 samples measured every 30 seconds for 15 minutes in situ, measured 5 minutes ex situ by LISST and measured 5 times per sample by Mastersizer (for all different stirring speeds and storage durations).

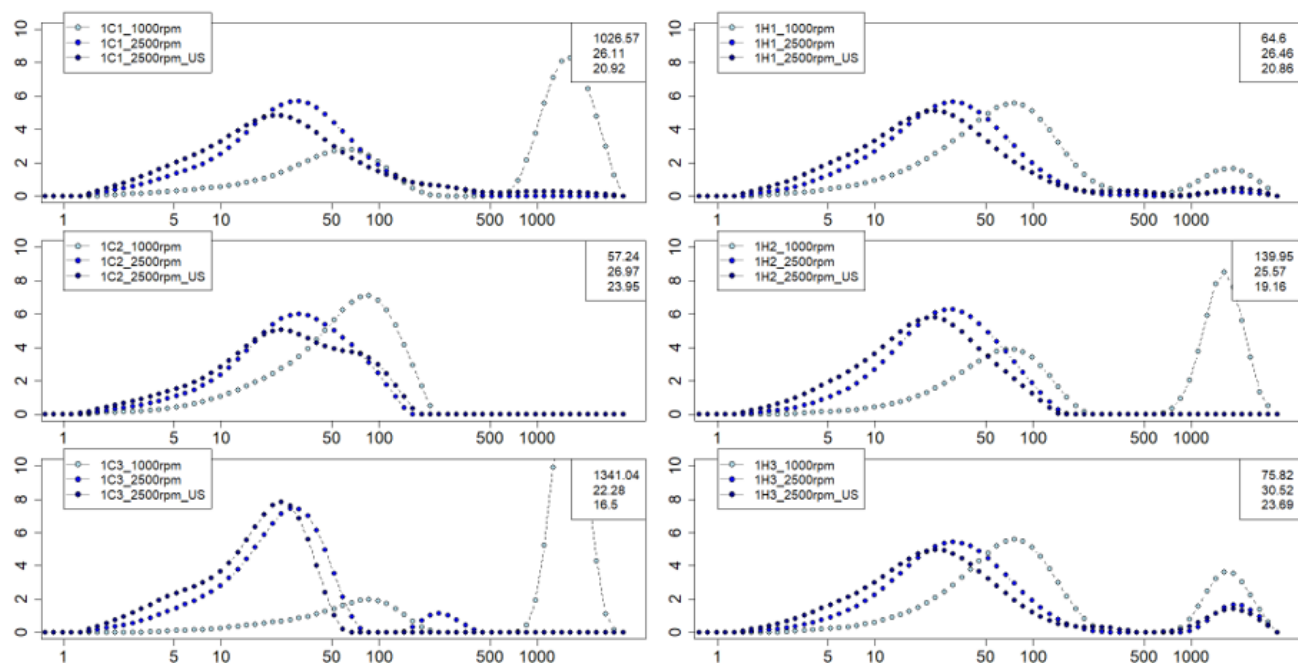


Figure S3. Ex situ particle size distributions of sample 1 measured by Mastersizer at three stirrer settings (1000 rpm, 2500 rpm, 2500 rpm + US) stored for 3 weeks, including the corresponding D₅₀ values. Left panel: cold storage (C) and right panel: hot storage (H).

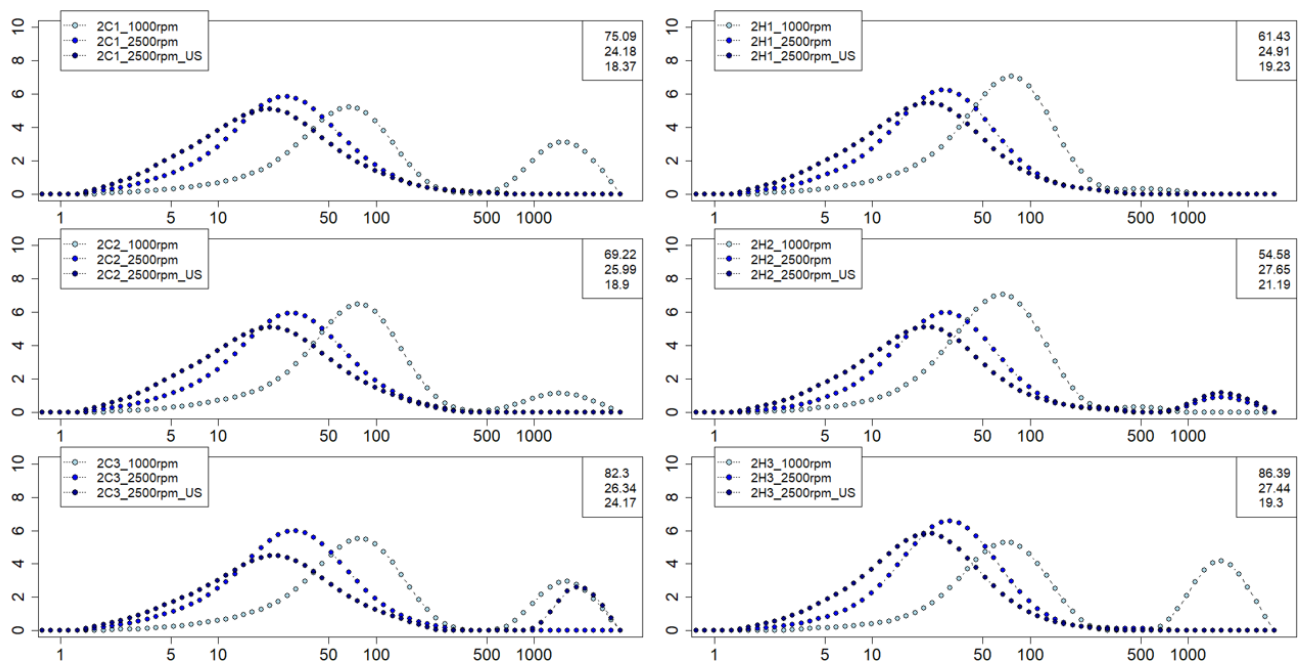


Figure S4. Ex situ particle size distributions of sample 2 measured by Mastersizer at three stirrer settings (1000 rpm, 2500 rpm, 2500 rpm + US) stored for 3 weeks, including the corresponding D_{50} values. Left panel: cold storage (C) and right panel: hot storage (H).

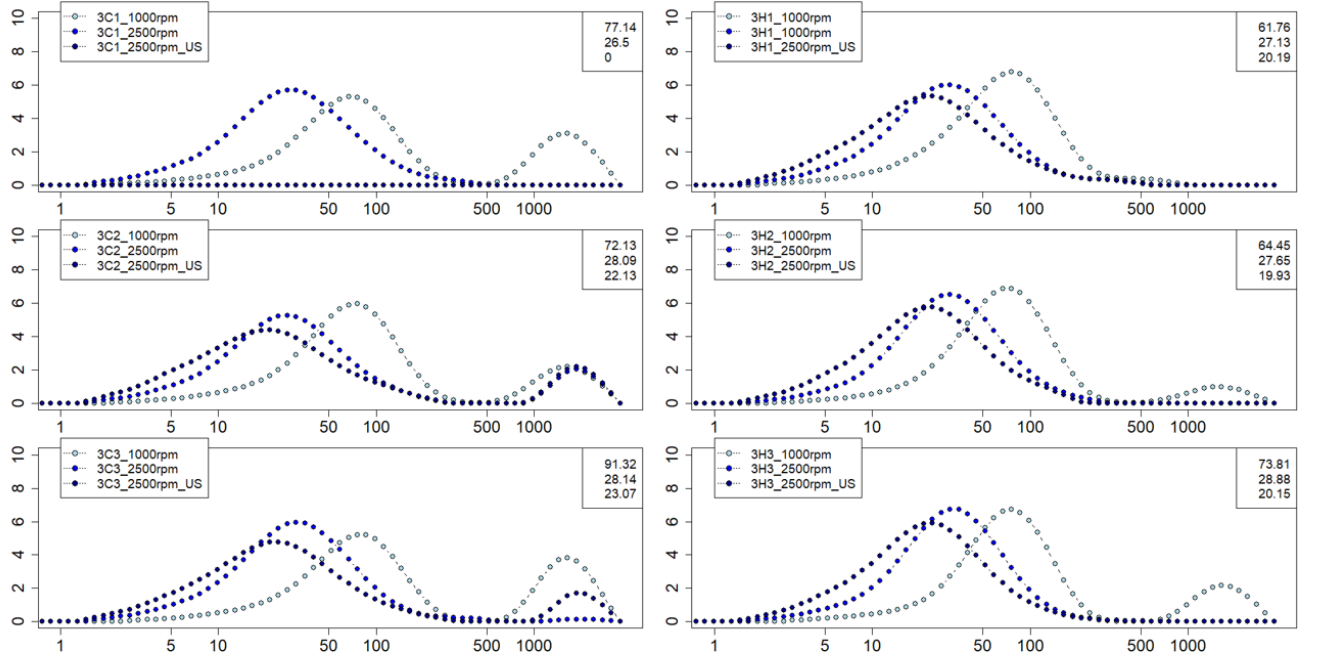


Figure S5. Ex situ particle size distributions of sample 3 measured by Mastersizer at three stirrer settings (1000 rpm, 2500 rpm, 2500 rpm + US) stored for 3 weeks,, including the corresponding D_{50} values. Left panel: cold storage (C) and right panel: hot storage (H).

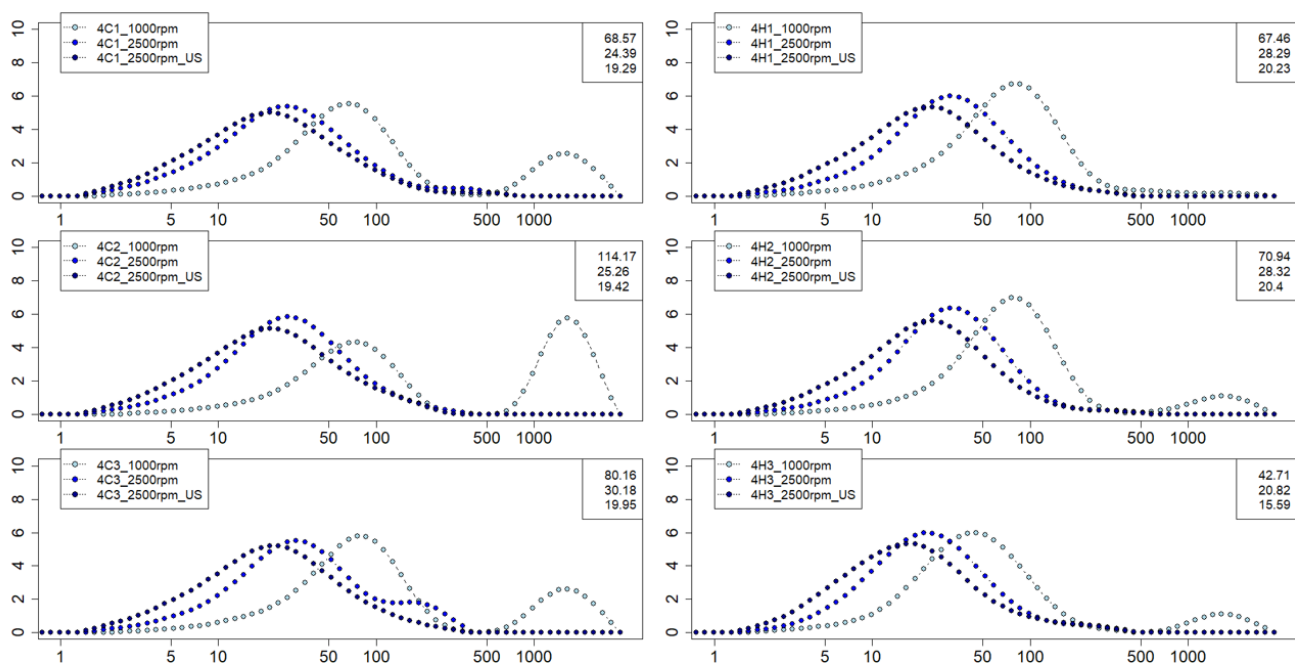


Figure S6. Ex situ particle size distributions of sample 4 measured by Mastersizer at three stirrer settings (1000 rpm, 2500 rpm, 2500 rpm + US) stored for 3 weeks, including the corresponding D_{50} values. Left panel: cold storage (C) and right panel: hot storage (H).

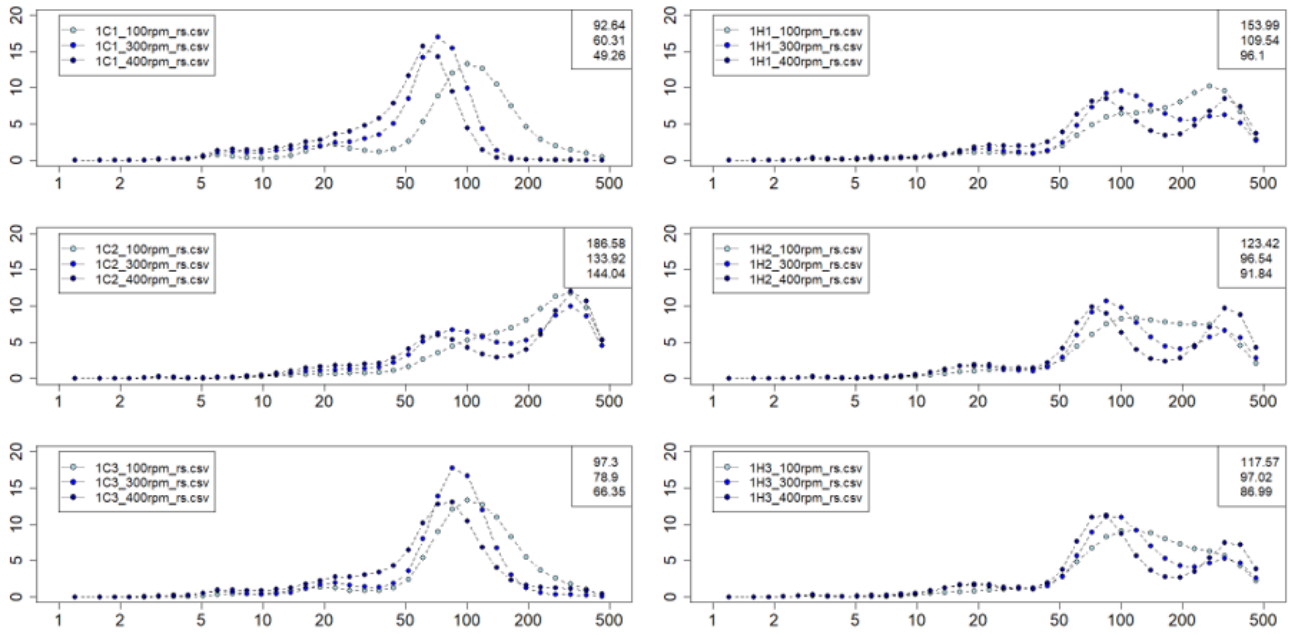


Figure S7. Ex situ particle size distributions of sample 1 measured by LISST at three stirrer settings (100 rpm, 300 rpm, 400 rpm) stored for 3 weeks, including the corresponding D_{50} values. Left panel: cold storage (C) and right panel: hot storage (H).

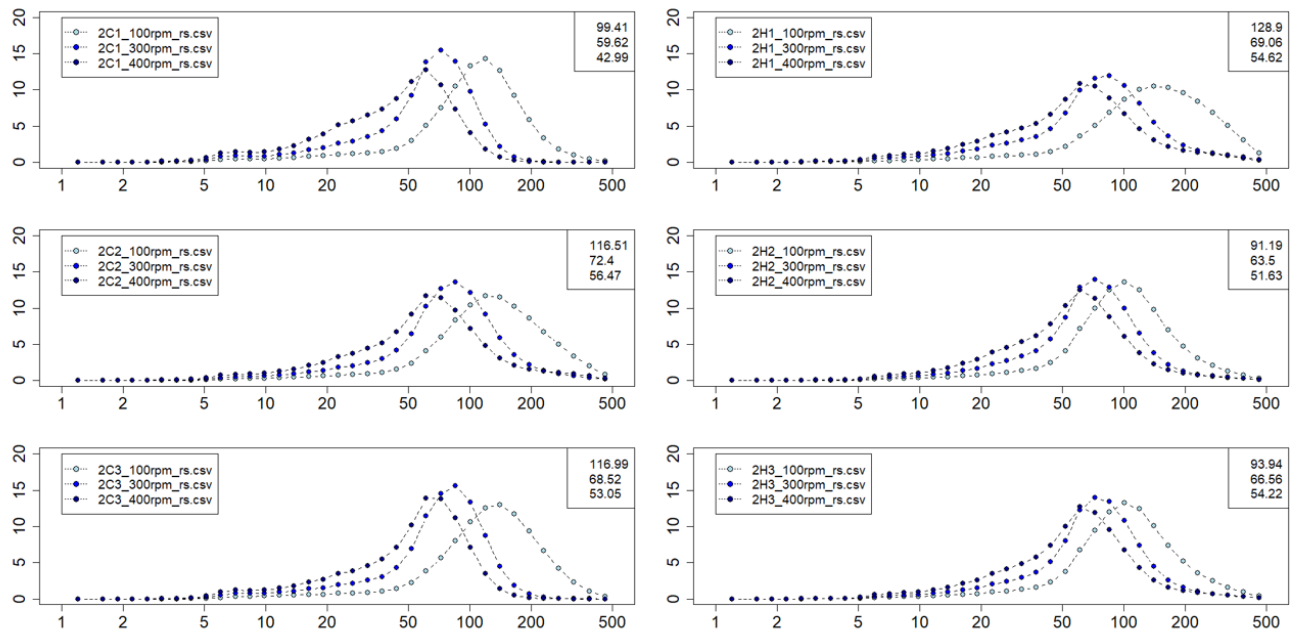


Figure S8. Ex situ particle size distributions of sample 2 measured by LISST at three stirrer settings (100 rpm, 300 rpm, 400 rpm) stored for 3 weeks, including the corresponding D_{50} values. Left panel: cold storage (C) and right panel: hot storage (H).

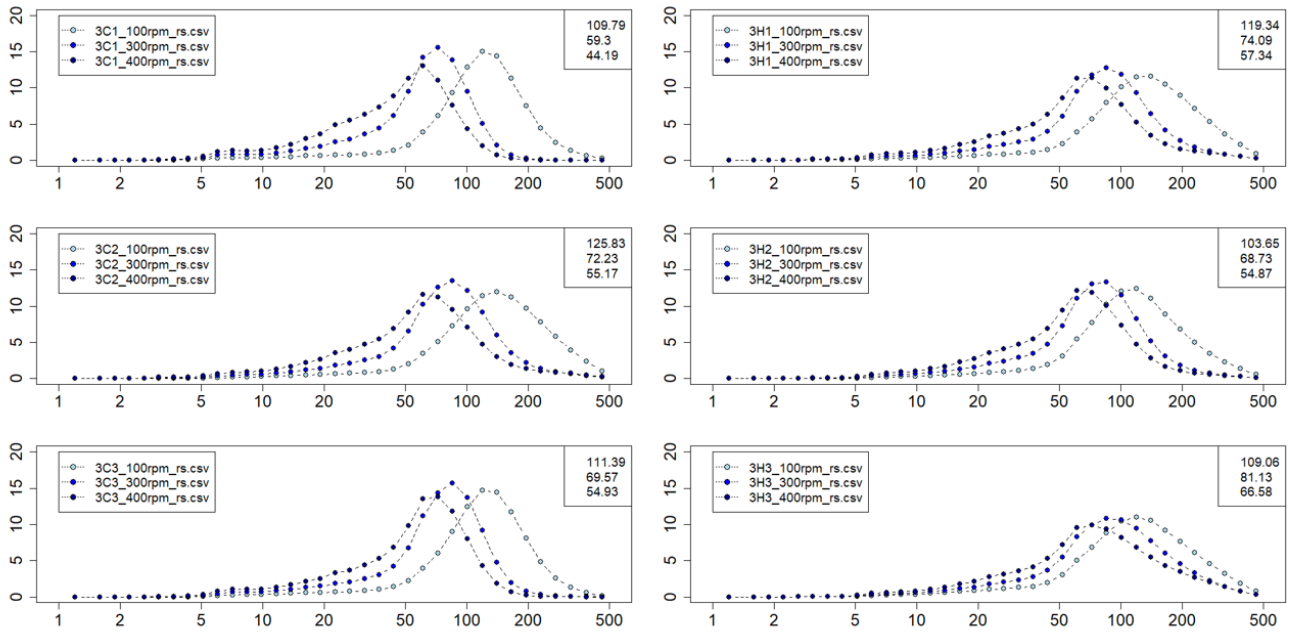


Figure S9. Ex situ particle size distributions of sample 3 measured by LISST at three stirrer settings (100 rpm, 300 rpm, 400 rpm) stored for 3 weeks, including the corresponding D_{50} values. Left panel: cold storage (C) and right panel: hot storage (H).

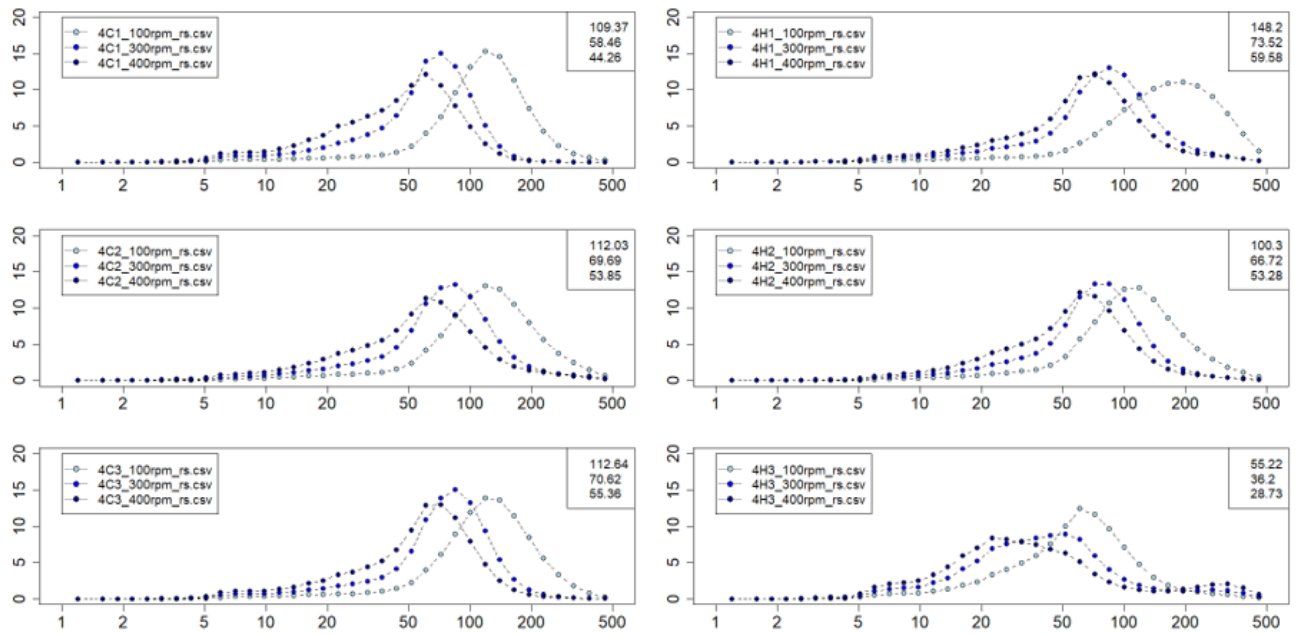


Figure S10. Ex situ particle size distributions of sample 4 measured by LISST at three stirrer settings (100 rpm, 300 rpm, 400 rpm) stored for 3 weeks, including the corresponding D_{50} values. Left panel: cold storage (C) and right panel: hot storage (H).

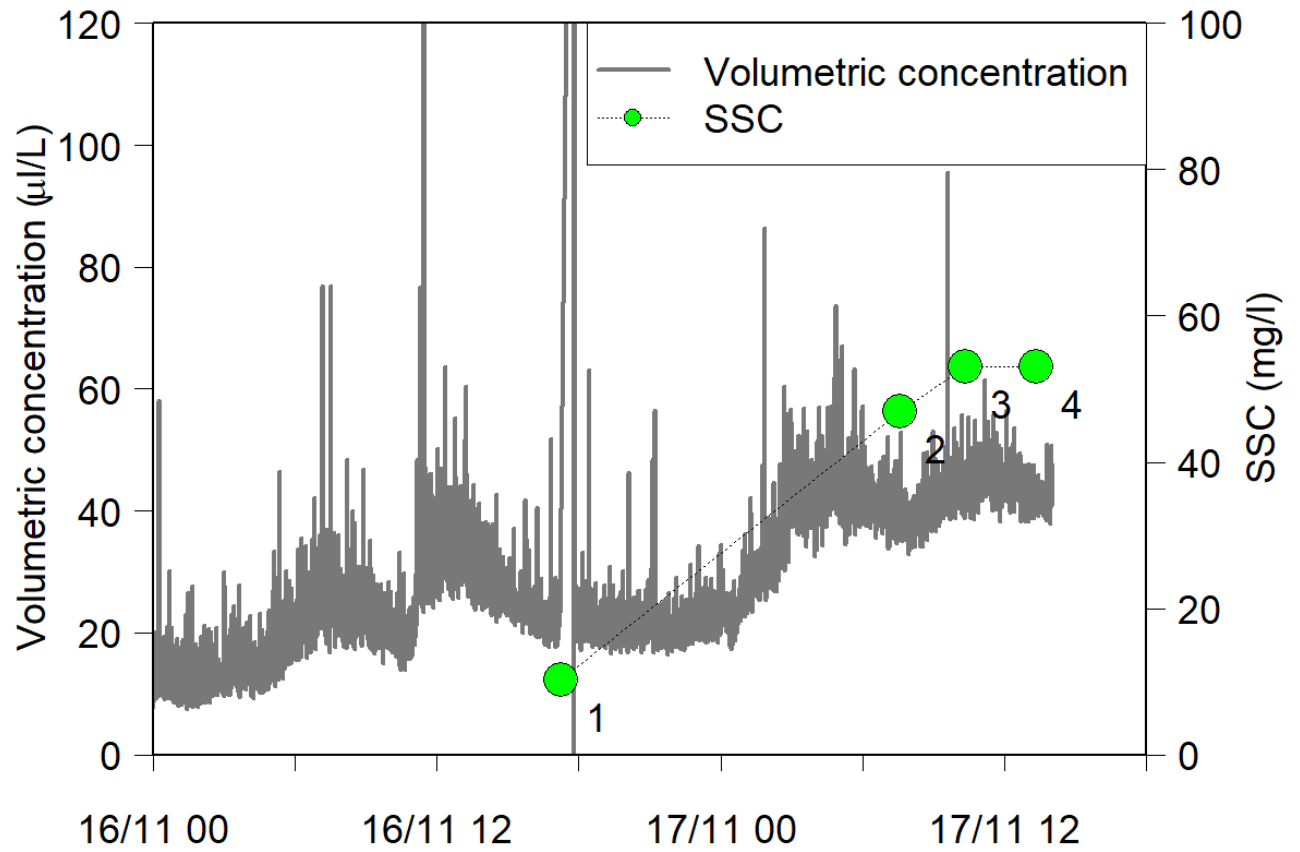


Figure S11. Volumetric concentration measured with LISST-200X during the rising limb of a rainfall-runoff event between 16/11/2022 - 18/11/2022. In green the gravimetric suspended particulate matter concentration of the four samples.

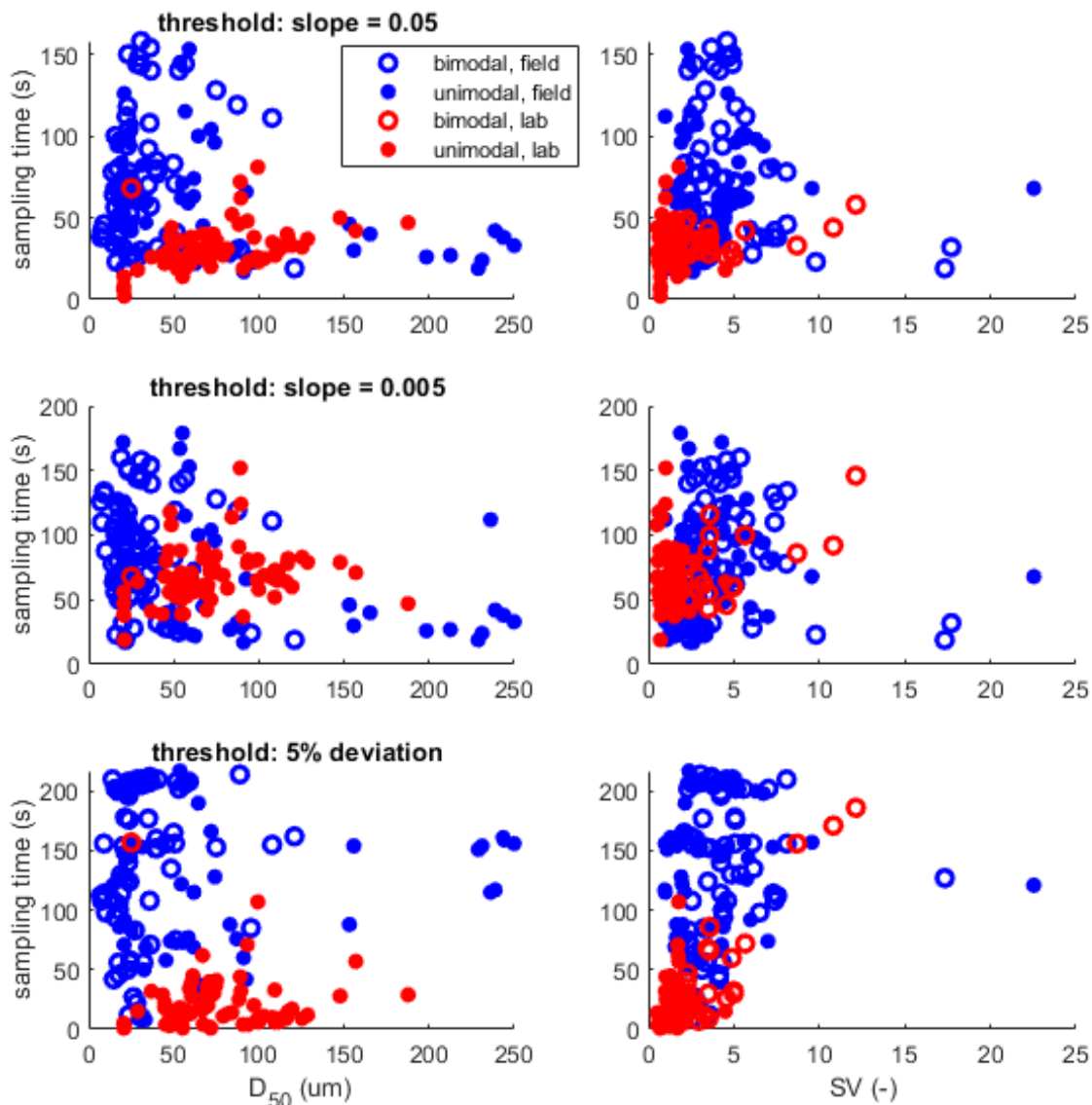


Figure S12. Relation between D_{50} (left) or SV (right) and sampling time, using three different thresholds. Field samples are indicated in blue, lab samples in red. Open circles are bi- or multimodal samples, closed circles are unimodal samples.

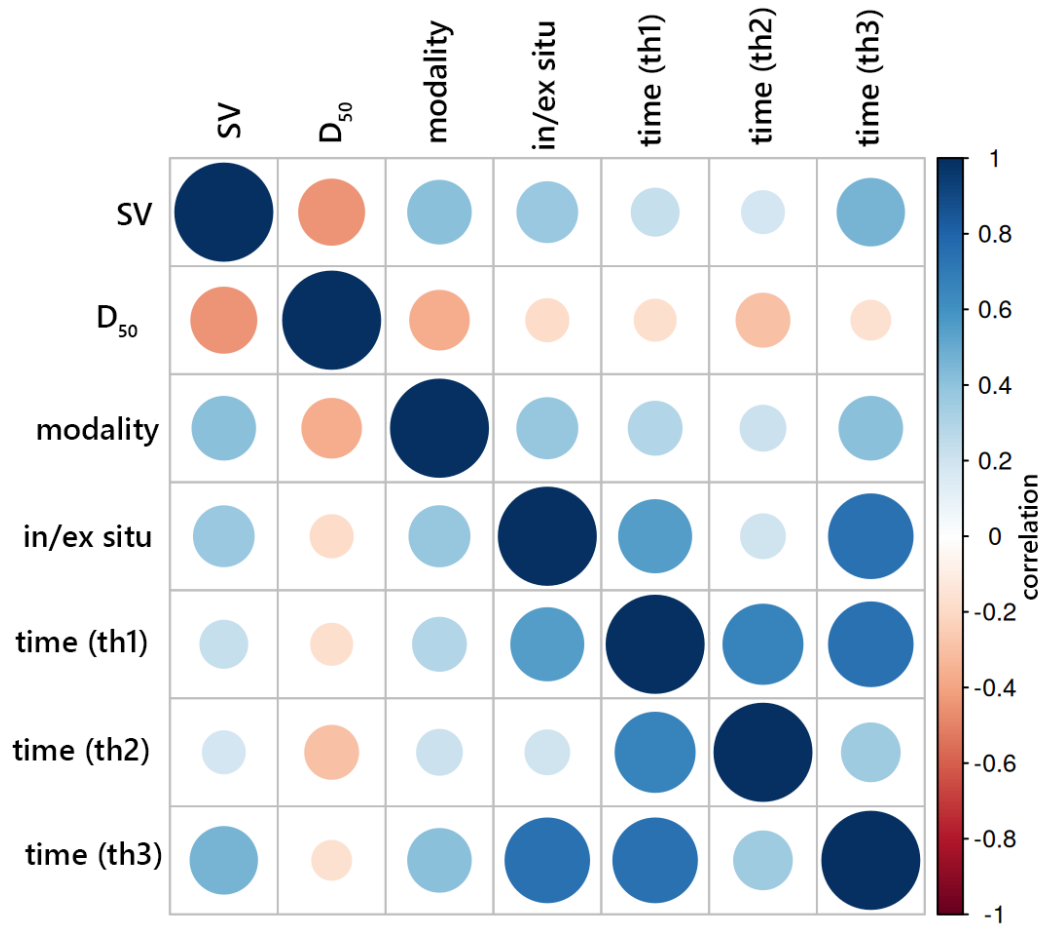


Figure S13. Linear correlations between the PSD characteristics and the required measurement times using three different thresholds (th)

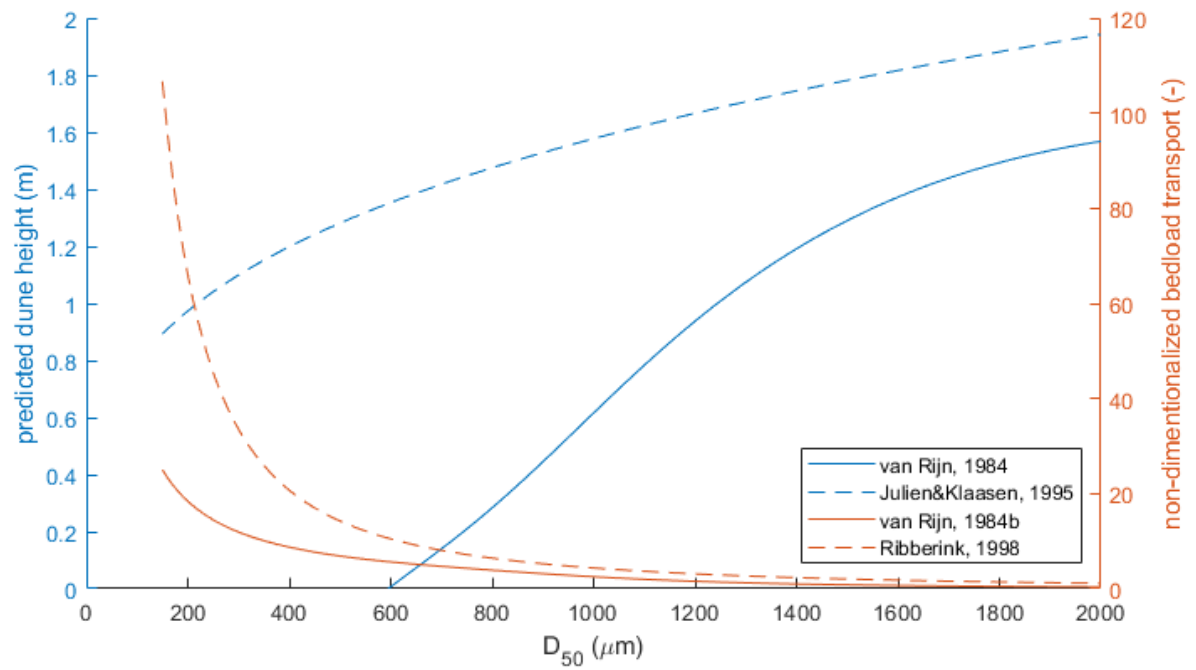


Figure S14. The sensitivity of two dune height predictors (in blue) L. C. van Rijn (1984); Pierre and Klassen (1995) and two sediment transport predictors (in orange) L. van Rijn (1993); Ribberink (1998) for grain size. Example with a water depth of 10 m, slope of 0.0001 and a water temperature of 15°C.

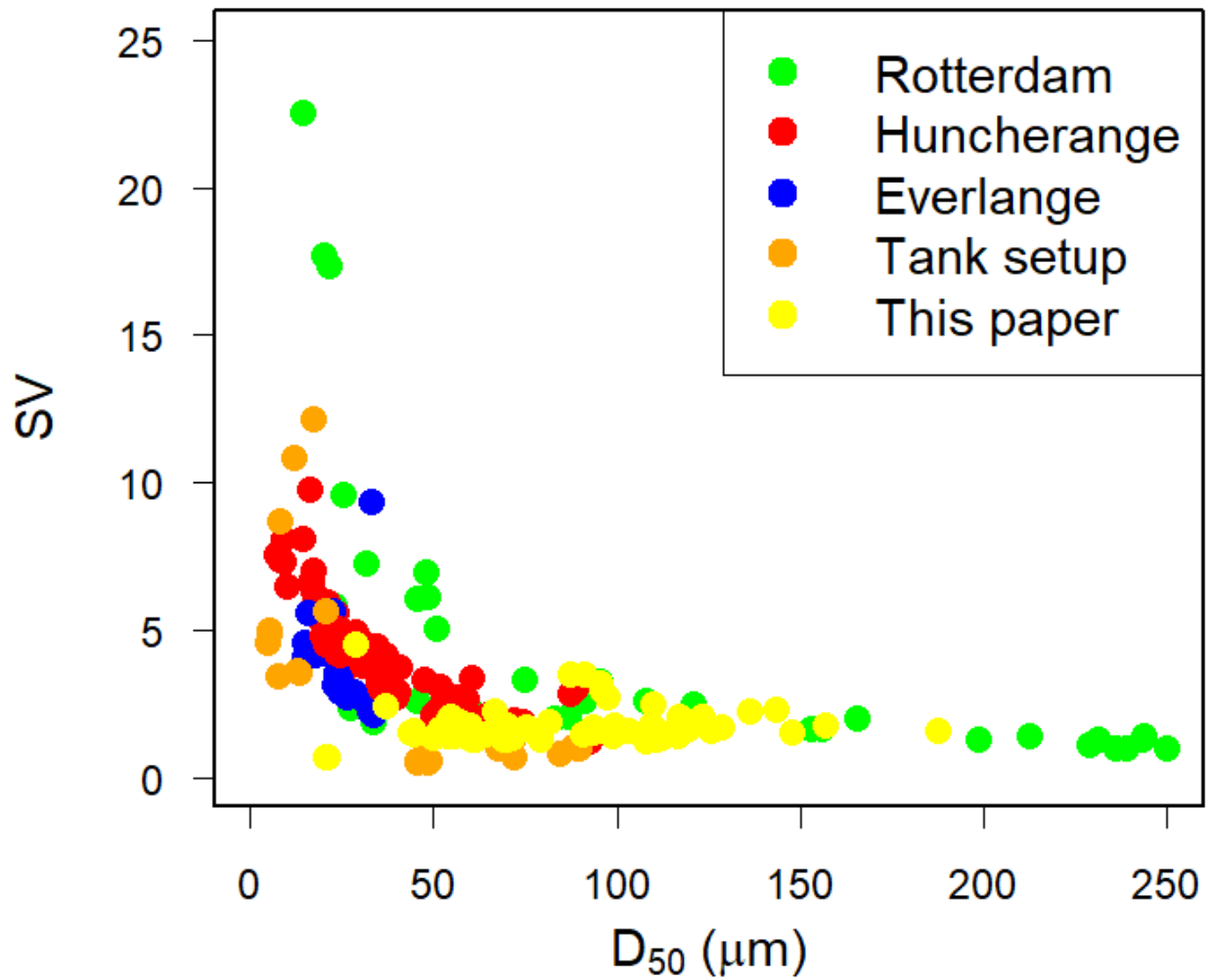


Figure S15. The relation between median particle size (D_{50}), span value (SV), and data set (colours).

Sample		ex situ LISST									
In situ LISST	Speed	Cold			Hot			Mean			
		Week 1	Week 2	Week 3	Week 1	Week 2	Week 3	Mean	Std		
1	100	92.6	186.6	97.3	154.0	123.4	117.6	128.6	32.7		
	300	60.3	133.9	78.9	109.5	96.5	97.0	96.0	23.1		
	400	49.3	144.0	66.3	96.1	91.8	87.0	89.1	29.4		
	100	99.4	116.5	117.0	128.9	91.2	93.9	107.8	13.8		
2	300	59.6	72.4	68.5	69.1	63.5	66.6	66.6	4.1		
	400	43.0	56.5	53.0	54.6	51.6	54.2	52.2	4.4		
	100	109.8	125.8	111.4	119.3	103.7	109.1	113.2	7.3		
	300	59.3	72.2	69.6	74.1	68.7	81.1	70.8	6.6		
3	400	44.2	55.2	54.9	57.3	54.9	66.6	55.5	6.5		
	100	109.4	112.0	112.6	148.2	100.3	55.2	106.3	27.3		
	300	58.5	69.7	70.6	73.5	66.7	36.2	62.5	12.7		
	400	44.3	53.8	55.4	59.6	53.3	28.7	49.2	10.2		

Sample	In situ LISST	ex situ Mastersizer								
		Speed	Cold			Hot			Mean	
			Week 1	Week 2	Week 3	Week 1	Week 2	Week 3	Mean	Std
1	26.2	1000	1026.6	57.2	1341.0	64.6	140.0	75.8	93.5	40.7
		2500	26.1	27.0	22.3	26.5	25.6	30.5	26.3	2.4
		2500+US	20.9	23.9	16.5	20.9	19.2	23.7	20.8	2.6
		1000	75.1	69.2	82.3	61.4	54.6	86.4	71.5	11.1
2	47.6	2500	24.2	26.0	26.3	24.9	27.6	27.4	26.1	1.2
		2500+US	18.4	18.9	24.2	19.2	21.2	19.3	20.2	2.0
		1000	77.1	72.1	91.3	61.8	64.5	73.8	73.4	9.6
		2500	26.5	28.1	28.1	27.1	27.7	28.9	27.7	0.8
3	52.9	2500+US	NA	22.1	23.1	20.2	19.9	20.1	21.1	1.3
		1000	68.6	114.2	80.2	67.5	70.9	42.7	74.0	21.3
		2500	24.4	25.3	30.2	28.3	28.3	20.8	26.2	3.1
		2500+US	19.3	19.4	19.9	20.2	20.4	15.6	19.1	1.6
4	52.5	2500	24.4	25.3	30.2	28.3	28.3	20.8	26.2	3.1
		2500+US	19.3	19.4	19.9	20.2	20.4	15.6	19.1	1.6

Table S1. D_{50} values of all in and ex situ measurements with the LISST-200X and Mastersizer-3000. for all storage

durations and stirring speeds.